

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	HILGREN ET AL.	Examiner:	J. PAK
Serial No.:	09/614,631	Group Art Unit:	1653
Filed:	JULY 12, 2000	Docket No.:	163.1382US01
Title:	METHOD AND COMPOSITION FOR INHIBITION OF MICROBIAL GROWTH IN AQUEOUS FOOD TRANSPORT AND PROCESS STREAMS		

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

I, John D. Hilgren, declare and state the following:

1. I am an inventor of the subject matter of the patent application identified above and an employee of Ecolab, Inc., the assignee of the patent application identified above.

2. I understand that the Examiner has cited Hei (US 6,024,986) as prior art in prosecution of the application identified above. I understand that the Hei patent was filed on May 24, 1999.

3. I further understand that the filing date of the present patent application Serial No. 09/614,631 is July 12, 2000.

4. I state that before the date of the Hei patent, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. We made this invention in the United States.

5. The claims of the patent application identified above relate to compositions including:

about 35 to about 45 weight-% acetic acid, about 5 to about 15 weight-% octanoic acid, about 3 to about 8 weight-% hydrogen peroxide, about 8 to about

16 weight-% peroxyacetic acid, about 1 to about 5 weight-% peroxyoctanoic acid, and about 0.1 to about 2 weight-% chelating agent;

about 40 weight-% acetic acid, about 10 weight-% octanoic acid, about 5 weight-% hydrogen peroxide, about 12 weight-% peroxyacetic acid, about 3 weight-% peroxyoctanoic acid, and about 0.6 weight-% chelating agent;

about 10 to about 150 ppm acetic acid, about 5 to about 40 ppm octanoic acid, about 4 to about 20 ppm hydrogen peroxide, about 5 to about 50 ppm peroxyacetic acid, about 2 to about 25 ppm peroxyoctanoic acid, and about 0.2 to about 2.5 ppm chelating agent;

about 133 ppm acetic acid, about 33 ppm octanoic acid, about 17 ppm hydrogen peroxide, about 40 ppm peroxyacetic acid, about 10 ppm peroxyoctanoic acid, and about 2 ppm chelating agent;

about 50 to about 60 weight-% acetic acid, about 10 to about 20 weight-% octanoic acid, about 5 to about 15 weight-% hydrogen peroxide, and about 0.3 to about 1 weight-% chelating agent; or

about 54 weight-% acetic acid, about 14 weight-% octanoic acid, about 10 weight-% hydrogen peroxide, and about 0.6 weight-% chelating agent;

Each claimed composition also has at least about 1 part by weight of peroxyoctanoic acid for each about 5 parts of peroxyacetic acid.

The present patent application includes at page 15 several tables describing embodiments of the claimed compositions. Two of the tables describe concentrate and use compositions including:

Chemical	Wt-%	ppm
Acetic Acid	40	133
Hydrogen Peroxide	5	17
HEDP	0.6	2
Octanoic Acid	10	33
Peroxyacetic Acid	12	40
Peroxyoctanoic Acid	3	10

One of the tables describes raw materials that can be used to make the concentrate composition described above. These raw materials include:

Raw Material	Weight %
Glacial Acetic Acid	54
Hydrogen Peroxide, 35%	30
HEDP, 60%	1
Octanoic Acid, 95%	15

6. During the research and development leading to the claimed compositions, and before May 24, 1999, my coinventors and I made several compositions meeting the limitations of the claims of the present application and including the compositions described in the tables in paragraph 5 above.

7. Accompanying Exhibit A reports studies of compositions that meet the limitations of the present claims and including the compositions described in the Tables in paragraph 5 above. In Exhibit A, the initial, concentrate, and use compositions described in the tables in paragraph 5 above are referred to as "Falcon 15 O". The composition called "Falcon 15 AE" also meets the limitations of the present claims. The contents of these formulas can be found at page 9-10 of Exhibit A. Page 9 of Exhibit A describes the stability of these formulas. The pages of the report attached as Exhibit A were prepared before May 24, 1999.

8. The evidence presented in Exhibit A indicates that, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. Moreover, Exhibit A demonstrates the concentrations of the compositions called Falcon 15 O and Falcon 15 AE, as invented prior to May 24, 1999, fit within the claims of the present invention. Pages 1 and 9 of exhibit A also demonstrates the compositions of Falcon 15 invented prior to May 24, 1999, were physically stable.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

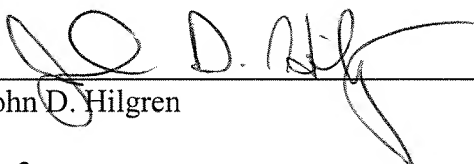

 John D. Hilgren

EXHIBIT A
DATED MAY 25, 2006
APPLICATION NO. 09/614,631

W-299

Work continued from Page

OBJECTIVE: Develop a concentrated version of Falcon KX-6049. Or develop a sanitizer concentrate that maximizes actives (POAA, POOA, H₂O₂) while minimizing cost.

Discussion w/ [redacted]: [redacted] has developed a "Falcon 15" formula that is physically stable.

Formula	AM	#Equilibrium
Glacial Acetic Acid	59.0%	50.87
H ₂ O ₂ (35%)	25.0	3.50
Dequest 2010 (60% Active)	1.0	0.60
Octanoic Acid	15.0	12.28
POAA POAA	0.0	10.30
POOA	0.0	3.02
Water	0.0	19.67

*The Equilibrium concentrations were based on the "SOLVER" computer program. We chose equilibrium constants based on 3 samples of Falcon 15 produced analyzed by analytical. $K_1 = 1.7$ & $K_2 = 2.35$

FALCON 15 EQUILIBRIUM CONSTANTS DERIVATION

RICHTER/REINHARDT FORMULA

	% Raw Mat'l
Acetic Acid	59.00
Hydrogen Peroxide	8.75
Dequest 2010 (60%)	0.60
Octanoic Acid	15.00
POAA	
POOA	
Water	16.65
	100.00

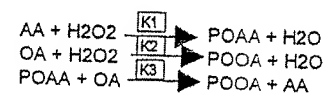
1.0443		
Concentration in Molarity		
9807-48	9889-36	9889-46
10.27	10.27	10.27
2.69	2.69	2.69
1.09	1.09	1.09
9.66	9.66	9.66
8.98	8.04	7.78
1.02	0.99	1.01
0.87	0.78	0.76
1.34	1.43	1.39
0.30	0.20	0.19
10.56	14.88	16.16

ANALYTICAL RESULTS

	9807-48	9889-36	9889-46
AAeq	8.98	8.04	7.78
H2O2eq	1.02	0.99	1.01
OAeq	0.87	0.78	0.76
POAAeq	1.34	1.43	1.39
POOAeq	0.30	0.20	0.19
H2Oeq	10.56	14.88	16.16

using these K_{eq}'s.
 **After Manipulation of SOLVER Program we decided:
 $K_1 = 1.7$ & $K_2 = 2.35$
 $K_1 = 2.77$ calculated based on Analytical
 $K_2 = 4.00$

2 Week Equilibrium Concentrations (% By Weight)				
	9807-48	9889-36	9889-46	SOLVER
	51.57	46.19	44.70	50.87
	3.33	3.21	3.29	3.50
	0.60	0.60	0.60	0.60
	12.00	10.80	10.50	12.28
	9.73	10.41	10.10	10.30
	4.57	3.14	2.95	3.02
	18.20	25.65	27.86	19.67
	100.00	100.00	100.00	100.24



SIGNATURE

K1 =	1.54	2.69	2.85
K2 =	3.54	3.95	4.05
K3 (from eq)	2.30	1.47	1.42

K1 ave of last two:	2.77
K2 ave of last two:	4.00
K3 ave of last two:	1.44

10-01-16

Work continued from Page 34

According to [REDACTED], original goal was to maximize octanoic acid content in raw material but they determined a solubility limit near 15%. Physical stability tests are performed at 40°F and 100°F.

It appears from Micro testing that the lower the H_2O_2 the better. We will focus on ① Minimizing cost ② maintain physical stability ③ maintain chemical stability ④ maximize PA/PA+ H_2O_2 ratios ⑤ Understand other factors involved that increases efficacy. ⑥ Make Non-Detonable Solutions.

I. Physical Stability TESTS

A) CONCENTRATE

- ① Room Temp.
- ② 40°F
- ③ 100°F - 1 MONTH?

B) Use Solution - May use

- ① Room Temp
- ② 40°F
- ③ 100°F

Analytical G.C.
To determine if octanoic separates to the top.

- I. Qualitative - Turbidity
- II. Quantitative - G.C

II. Chemical Stability TESTS

- CONCENTRATE ONLY

- ① Room Temp.
- ② 100°F

Method of Analysis

- ① QATM 203
- ③ HPLC

III. EFFICACY

- ① - At use concentrations
- 4 exposure times / Temp. / Etc.

SIGNATURE [REDACTED]

DATE [REDACTED]

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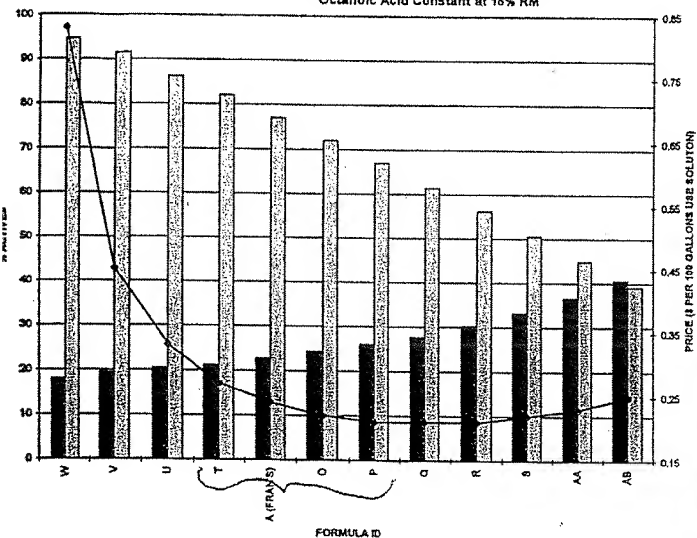
DATE [REDACTED]

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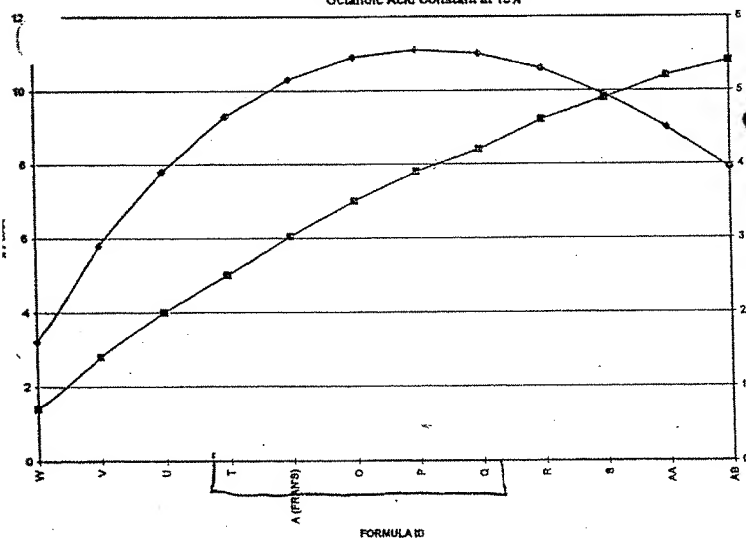
Work continued from Page 35

Important Areas of INTEREST WHEN DECIDING UPON A FALCON 15 FORMULA:

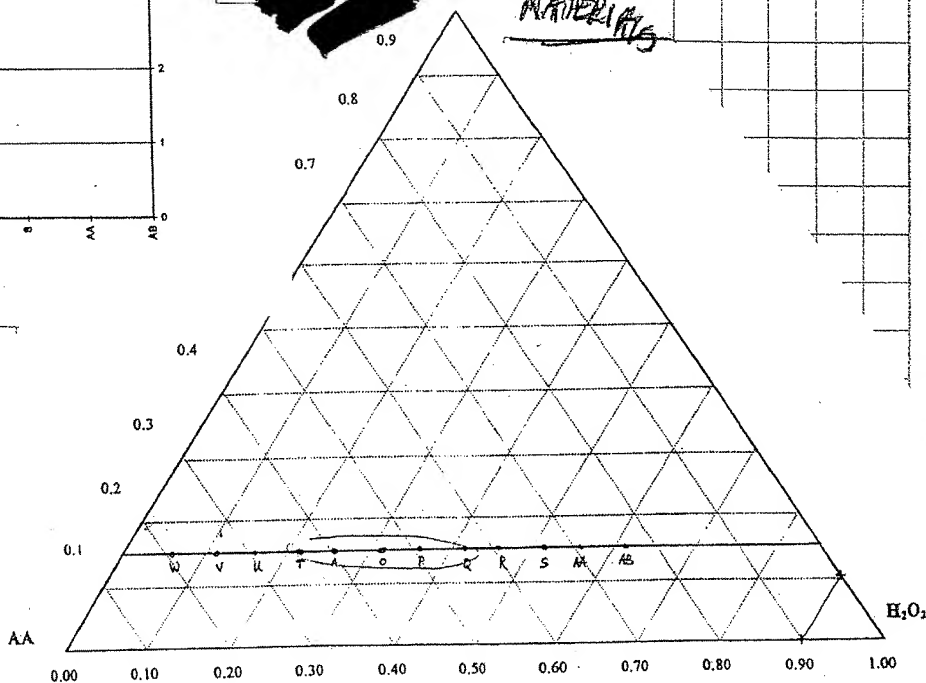
FALCON 15 POTENTIAL FORMULAS
Octanoic Acid Constant at 15% RM

	AA	H2O2
W	78%	5%
V	74%	10%
U	68%	15%
T	64%	20%
A (FRAN)	58%	25%
O	54%	30%
P	48%	35%
R	44%	40%
S	38%	45%
AA	34%	50%
AB	28%	55%
AB	24%	60%

* For Ternary diagram, determine where the detonable formulas are and where the unstable (physically) formulas are.

FALCON 15 POTENTIAL FORMULAS
Octanoic Acid Constant at 15%

RAW MATERIALS



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Work continued from Page 36

Physical Stability TESTS - MIX ALL SAMPLES
 VIAL A FALCON 15 FORMULA FOR 5 minutes - Stir Plate

CONCENTRATION	TEMP.	QUALITATIVE ANALYSIS
FALCON 15 - CONCENTRATE Prepared by Joel Schilling 9/19/96 SG = 1.0443	100°F	START DATE: 12-27-96
102/13 gallons 601 ppm (V/V) Falc = 0.5815 gms 628 (W/W) H ₂ O = 925.3 gms	Room Temp 60°F	MIX w/ Tap H ₂ O / At small amount of oil drops, seemed to be on the surface, but after mixing not as evident
1202 ppm (V/V) Falc = 1.0175 1235 ppm (W/W) H ₂ O = 810.2	Room Temp 60°F	ONCE mixed appears stable
1803 (V/V) Falc = 1.9075 1883 (W/W) H ₂ O = 958.4	Room Temp 60°F	An obvious oil separation on top before thoroughly mixing. After mixing very slight oil droplets.
2232 (V/V) Falc = 1.9020 2331 (W/W) H ₂ O = 815.2	Room Temp 60°F	Oil separation on top before mixing. *obvious oil slick after mixing
6695 (V/V) Falc = 5.610 6992 (W/W) H ₂ O = 798.2	Room Temp 60°F	Heavy oil slick prior to mixing and after mixing
9576 (V/V) Falc = 8.1305 1% (W/W) H ₂ O = 805.1	Room Temp 60°F	Heavy oil slick before and after mixing.

(SEE PP. 40)

* Appears that solubility limit for this Falcon 15 () formula is ~~between~~ around 1800 ppm (V/V) at 60°F.

* Could we use a H₂O soluble dye to stand out greater?

$$\frac{10^6}{(X \text{ ppm})(128)} = X \text{ gallons for 1 oz}$$

9980

Work continued from Page

Objective: Determine if chemical stability changes substantially over time at elevated temperatures.

SOLUTION: [REDACTED] Falcon 15 prepared [REDACTED]

Analytical Results from [REDACTED]:

FORMULA

59% - AA
2.5% - H_2O_2 (35%) - 8.75% (100%)
15% - OA
1% - Degrease 2010

OA - 8.73%
POOA - 2.67%
 H_2O_2 - 4.12%
POAA - 12.30%

LC

Analytical Results

ANALYTICAL

ANALYTICAL RESULTS

Room Temp
10/3/96
COMMENTS

Room Temp
Start of Test
([REDACTED])

Room Temp
End of 1 month
([REDACTED])

100°F
End of 1 month
([REDACTED])

Acetic Acid

N/A

59.80 50.67

51.22

63.0%

 H_2O_2

4.12%

3.20 3.20

1.50

<0.1%

POAA

11.03%

10.20 9.13

7.88

0.40%

POOA

2.67%

2.26 2.26

2.24

0.27%

OA

8.73%

12.20 12.20

13.0

18.1%

0.6% Degrease 0.6% Deg.
11.74% H_2O 21.94% H_2O

OVEN START DATE: [REDACTED]

END DATE: [REDACTED]

SIGNATURE [REDACTED]

DATE [REDACTED]

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DATE [REDACTED]

WITNESS [REDACTED]

DATE [REDACTED]

Determine upper OA limit

BOOK NO.

9980

Work continued from Page

Objective: Prepare the following Falcon 15 formulas to determine physical stability of high OA concentrations

FORMULA

Room Temp
Stability40°F
Stability (if Room
Temp is stable)

X - 57% - AA - 41.04 gms
20% - H₂O₂ - 13.95
20% - OA - 13.98
1% - Dequest - 0.74

STABLE CLEAR
SOLUTIONSTABLE CLEAR
SOLUTION

Y - 47% - AA - 34.53 gms
30% - H₂O₂ - 21.18
20% - OA - 14.10
1% - Dequest - 0.71

Using 40°F H₂O₂ - When
mixing was not stable,
but once at room
temp. became stable
& clear.

NOT STABLE
Separated into two
layers.

Z - 39% - AA - 26.89 gms
40% - H₂O₂ - 21.18
20% - OA - 14.10
1% - Dequest - 0.71

NOT STABLE - Top
Octanoic Acid Layer.Did not test -
No need to.

* Depending on stability or instability of these formulas we will
vary OA concentration.

Glacial Acetic Acid - 124016 LOT #115371

35% H₂O₂ - 240317 Lot B5762-144

Octanoic Acid - 190280 Lot 2176/55

Dequest ADPA - 60AW 250282 Lot# 05086-0580DR

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Work continued to Page 40

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Determine Upper OA Limit

BOOK NO.

9980

Work continued from Page 39

According to "Merck Index" Octanoic Acid has a maximum solubility of 0.068 gms / 100 gms H₂O or (680 ppm w/w). It has greater solubility in acetic acid.

680 ppm (w/w) octanoic acid would be equivalent to

$$\frac{680}{(1.0943)(1.1228)} = 5,303 \text{ ppm (v/v) Falcon 15 @ } 25^{\circ}\text{C or Room Temperature.}$$

\uparrow \uparrow
 S.G. % Octanoic Acid
 by "Solvent"

* We noticed solubility problems at 1800 ppm (v/v) although this was at 16°C or 60°F.

* At room temp. we would expect to see slightly better solubility than 5303 due to the coupling ability of acetic acid. Although, we are not sure of the solubility limit of peroctanoic acids. - most likely no limits.

* The samples from pp. 37 were also placed in 40°F refrigerator

All samples use [redacted] Falcon 15	STABILITY 40°F	STABILITY 60°F	STABILITY 70°F
601 ppm (v/v)	STABLE	STABLE - Actual	STABLE
1202	STABLE	STABLE - Raw	STABLE
1803	STABLE	UNSTABLE - Material	STABLE
2232	STABLE	UNSTABLE - Whole	STABLE
6695	UNSTABLE	UNSTABLE - Samples	UNSTABLE
9576	UNSTABLE	UNSTABLE	UNSTABLE

Sample of solution prepared

* NOTE: The 40°F stability tests were performed by sampling from a larger sample while mixing. The solution may have already separated. Repeat experiment with large sample

SIGNATURE [redacted]

DATE [redacted]

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DATE [redacted]

WITNESS [redacted]

DATE [redacted]

Work continued from Page 40

Additional Concentrations to narrow in on Formulas -
Determining Physical Stability.

FORMULA	Room Temp. STABILITY	40°F STABILITY
Q AA - 44% - 56.79 gms H ₂ O ₂ - 40% - 51.64 Dequest - 1% - 1.29 OA - 15% - 19.47	NOT STABLE	NOT STABLE
R AA - 39% H ₂ O ₂ - 45% Dequest - 1% OA - 15%	ASSUME INSTABILITY BASED UPON Q & the fact that AA is a coupler	
Y1 AA - 52% - 54.58 gms H ₂ O ₂ - 30% - 31.56 Dequest - 1% - 1.15 OA - 17% - 17.86	STABLE	NOT STABLE STABLE
Y2 AA - 49% H ₂ O ₂ - 33 Dequest - 1% OA - 17%	{ Assume Instability due to P & Y being unstable - did not prepare this sample.	
Z1 AA - 39% H ₂ O ₂ - 43% Dequest - 1% OA - 17%	{ Assume INSTABILITY due to Q being UNSTABLE	
Z2 AA - 42% H ₂ O ₂ - 40% Dequest - 1% OA - 17%	{ and the fact that AA is a coupler	

RAW MAT'L

PRODUCT LOT #S

* Glacial Acetic Acid: 124016 Lot #115371

Octanoic Acid: 90280 Lot 219605

35% H₂O₂: 240317 Lot B5762-144

Dequest ADPA: 250282 Lot 05056-0580 DR

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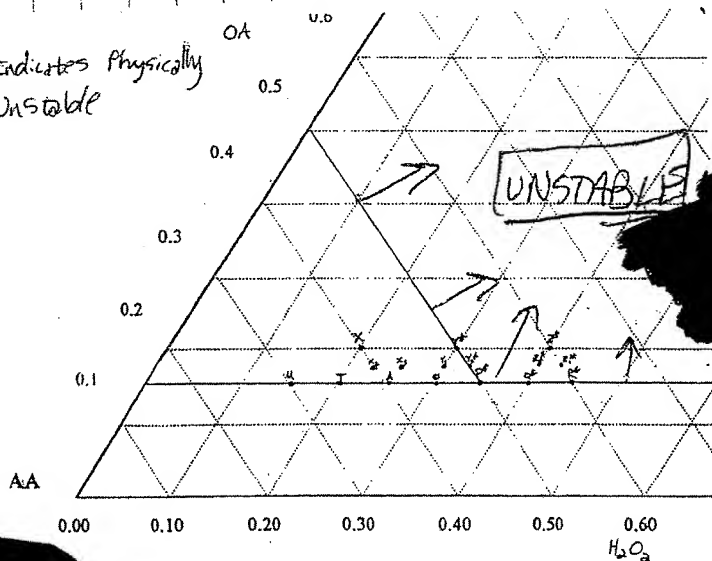
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work continued from Page A1

FORMULA	Room TEMP STABILITY	40°F STABILITY
P AA - 49% - 51.65 H_2O_2 - 35% - 37.0 Request - 1% - 1.05 OA - 15% - 15.81	UNSTABLE STABLE	UNSTABLE
X ₂ AA - 59% - H_2O_2 - 23% - Request - 1% - OA - 17% -	Assume STABILITY Based upon stability of X ₂ & X ₃	
X ₃ AA - 56% - 53.16 H_2O_2 - 26% - 24.0 Request - 1% - 1.00 OA - 17% - 16.24	STABLE	STABLE

*STABILITY CURVE THIS FOR:

*Indicates Physically
Unstable

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continued to Page 46

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DATE

WITNESS

DATE

Work continued from Page 45

* Based upon efficacy data from [REDACTED] [REDACTED] [REDACTED] -
lower H_2O_2 proves to be more efficacious.

FORMULA

Room Temp.
STABILITY40°F
STABILITY

AC: AA - 49.0 - 36.46 gm's
 H_2O_2 - 40.0 - 29.77
 Deguest - 1.0 - 0.75
 OA - 10.0 - 7.45

STABLE

UNSTABLE

AD: AA - 39.0 -
 H_2O_2 - 50.0 -
 Deguest - 1.0 -
 OA - 10.0 -

{ Did Not Prepare based upon
 AC - This will be unstable

AE: AA - 59.0 - 46.04
 H_2O_2 - 30.0 - 23.44
 Deg. - 1.00 - 0.82
 OA - 10.0 - 7.81

STABLE

STABLE

AF: AA - 69.0 - 48.51
 H_2O_2 - 20.0 - 14.03
 Deg. - 1.0 - 0.74
 OA - 10.0 - 7.04

STABLE

STABLE

A: AA - 59.0 - 39.21
 H_2O_2 - 25.0 - 16.63
 Deg. - 1.0 - 0.66
 OA - 15.0 - 10.00

STABLE

STABLE

O: AA - 54.0 - 32.57
 H_2O_2 - 30.0 - 18.11
 Deg. - 1.0 - 0.62
 OA - 15.0 - 9.05

STABLE

STABLE

T: AA - 64.0 - 43.31
 H_2O_2 - 20.0 - 13.54
 Deg. - 1.0 - 0.69
 OA - 15.0 - 10.16

STABLE

STABLE

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DATE

WITNESS

DATE

Work continued from Page 46

FORMULAS SENT TO ANALYTICAL FOR ANALYSIS:

	FORMULA ID	DATE Prepared	ANALYTICAL RESULTS				DATE:
			AA	H ₂ O ₂	POAA	POOA	
5							
10	"AF" AA - 69% H ₂ O ₂ - 20% OA - 10%		8.58	58.48	1.79	10.92	1.22
	"T" AA - 64% H ₂ O ₂ - 20% OA - 15%		11.2	53.28	1.89	10.92	1.45
15	"X" AA - 59% H ₂ O ₂ - 20% OA - 20%		15.8	47.49	1.82	10.11	2.72
	"A" AA - 59% H ₂ O ₂ - 25% OA - 15%		12.2	48.14	2.98	10.16	2.19
20	"AE" AA - 59% H ₂ O ₂ - 30% OA - 10%		7.45	46.64	3.98	13.46	1.76
25	"O" AA - 54% H ₂ O ₂ - 30% OA - 15%		11.4	40.99	4.15	13.11	2.51

NOTE: AF, T, A, AE & O - Had Equilibration @ room temp for 3 days
 X - " " " " " " 27 days

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DATE